Energy, Mines and Resources Canada | Energie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

ISO/TC 102/SC 2 - 13th INTERNATIONAL MEETING
TOKYO, JAPAN, 1986-11-9/14

R. Sutarno
Extractive Metallurgy Laboratory

December 1986

(For submission to the Standards Council of Canada)

Project: 3 0.6 9. 02
Analytical Methodology

10 pp

MINERAL SCIENCES LABORATORIES
DIVISION REPORT MSL 86-168 (TR)

ISO/TC 102/SC 2 - 13th INTERNATIONAL MEETING TOKYO, JAPAN, 1986-11-9/14

by

R. Sutarno*

This report covers the part of the proceedings of the 13th International Meeting of ISO/TC 102/SC 2 dealing with the work of ISO/TC 102/SC 2/WG 12B (Working Group on Evaluation of Statistical Data, Technical Committee 102 - Iron Ores, Subcommittee 2 - Chemical Analysis). Other topics discussed at the meeting will be reported by the leader of the Canadian delegation.

---

* Convener of ISO/TC 102/SC 2/WG 12B, Research Scientist, Physical Chemistry Section, Extractive Metallurgy Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, K1A 0G1.

ISO/TC 102/SC 2 - 13ième RÉUNION INTERNATIONALE TOKYO, JAPON, 1986-11-9/14

par

R. Sutarno*

Ce rapport présente la partie du procès-verbal de la 13ième réunion internationale du Groupe ISO/TC 102/SC 2 ayant pour objet les travaux du Groupe de travail sur l'évaluation des données statistiques, Comité technique 102 - Minerais de fer, Sous-comité 2 - Analyse chimique ISO/TC 102/SC 2/WG 12B. Les autres sujets discutés lors de la réunion feront l'objet d'un rapport qui sera présenté par le chef de la délégation canadienne.

*Président de ISO/TC 102/SC 2/WG 12B, Chercheur scientifique, Section de la chimie physique, Laboratoire de métallurgie extractive, Laboratoires des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, K1A 0G1.

C>

ISO/TC 102/SC 2/WG 12B MEETING

This meeting was called in order to discussed in detail various statistical problems faced by the sub-committee. This meeting was attended by small number of experts on statistics.

Date: 1986-11-09
Time: 1300 to 1700
Place: Shin-Sanya Club, Nippon Steel

Attendants:

E.S. Pilkington - Australia
M.G. Jecko - France
T. Miyazu - Japan
H. Umans - Netherlands
R. Sutarno - Canada (Convener)
A.C. Girodo - Brazil
T. Ohtsubo - Japan
T. Kondo - Japan
K. Takimoto - Japan
K. Mabuchi - Japan

Two topics were discussed in detail:

(1) Methods for presentation of precision data.

The precision data derived from the results of precision experiments (international tests) are presently presented in the standards in the form of linear regression equations as functions of the level of the characteristics to be determined. This method of presentation was considered to be an improvement from the tabular form previously used; it eliminates the boundary problem inherent in the tabular presentation. This method of presentation however, has its own disadvantages; among these are the problem of error of the regression equation itself. This error is generaly more prevalent when the concentration range is relatively wide. Occasionally the intercept of the regression line is negative. The reason for this may be that there is a difference in the chemical behaviour at various concentration levels that affects the measurement of the characteristic. Some suggestions were made at the meeting in an attempt to solve this problem. The group finally decided that the precision data will continue be presented in the form of a linear regression with the following modification:

(a). If the calculated value of a parameter (σ_r, σ_L, r or P) derived from the equation at the lowest experimental level (concentration tested) is less than the observed value, then the lowest experimental value should be used as the minimum value of the parameter.

(b). If the calculated value of the parameter derived from the equation at the lowest experimental level is greater than the observed value, then the regression equation should be altered such that the intercept of the line drawn between the two lowest experimental levels with the parameter axis or the average value of the parameters at these levels should be used as the intercept of the new regression line.

(2) Acceptability of analytical results reported by a laboratory.

In order to maintain the precision of the analytical results produced by the standard methods for analysis of iron ores, a certain procedure is required to be performed by the analysts. This procedure is illustrated schematically in Annex A of all standards issued by ISO/TC 102/SC 2 in recent years. This procedure requires the analyst to perform duplicate analyses and to test the results against the precision derived from the results of the precision experiment conducted for the particular method. Dr. Umans of the Netherlands felt however, that this procedure is inadequate and proposed a modified procedure. After lengthy discussion, the group decided to keep the present method (Annex A) unchanged. However, further discussion within the working group to improve this chart will be continued.

ISO/TC 102/SC 2 MEETING

Date:	1986-11-10/13
Place:	Japanese Iron & Steel Federation Keidanren Kaikan 1 chome 9-4, Ohtemachi Chiyoda-ku Tokyo 100,Japan
Attendants:	See official list of ISO/TC 102/SC 2 attendants (Appended to this report).

The work of WG 12B during the past period (1984/86) has been mainly to process all analytical data generated by working groups within the subcommittee.

Eight international tests were completed during the period; these are:

(a). Determination of vanadium content - Atomic absorption spectrometric method. Conducted by WG 18B.

(b). Determination of vanadium content - Spectrophotometric method. Conducted by WG 19B.

(c). Determination of nickel and chromium content - Atomic absorption spectrometric method. Conducted by WG 21B.

(d). Determination of carbon and sulfur content in DRI - High frequency/infrared method. Conducted by WG 24B.

(e). Determination of phosphorus content - Spectrophotometric method (Australian method). Conducted by WG 25B.

(f). Determination of manganese content - Atomic absorption spectrometric method. Conducted by WG 26B.

(g). Determination of total iron content - Wavelength dispersive X-ray fluoresence spectrometric method. Conducted by WG 27A.

(h). Determination of phosphorus content - Spectrophotometric method (Canadian method). Conducted by WG 28A.

The results of statistical evaluations of the above international test data have been communicated to the respective conveners and were used as one of the bases of discussion at the meeting. The results of these discussions were as follow:

(1). <u>The following methods were accepted in principle as standards and the working groups concerned were subsequently disbanded:</u>

(a). Determination of various elements content in iron ore - Wavelength dispersive X-ray fluorescence method.
ISO/TC 102/SC 2/WG 27B.

Note:
<u>Canada and Netherlands abstained and UK opposed this decision</u>

(b). Determination of vanadium content - Atomic absorption spectrometric method.
ISO/TC 102/SC 2/WG 18B.

(c). Determination of vanadium content - Spectrophotometric method.
ISO/TC 102/SC 2/WG 19B.

(d). Determination of nickel and chromium content - Atomic absorption spectrometric method.
ISO/TC 102/SC 2/WG 21B.

(e). Determination of phosphorus content - Spectrophotometric method.
ISO/TC 102/SC 2/WG 25B.

Note:
Due to the similarity of this method and the one developed by WG 28A, the best features of both methods have been incorporated; therefore it was not considered necessary to continue the work of WG 28A and the working group was subsequently disbanded.

(f). Determination of manganese content - Atomic absorption spectrometric method.
ISO/TC 102/SC 2/WG 26B.

(2). The development of the following methods has advanced to stage B and precision experiments will be conducted in the coming period:

(a). DRI - Determination of carbon and sulfur content - High frequency/infrared method.
ISO/TC 102/SC 2/WG 24A.

(3). The development of the following methods is still be at stage A:

ISO/TC 102/SC 2/WG 27A mandate will be extended for another two years to study the possibility of developing an X-ray fluoresence method for the determination of total iron content.

(4). The following new working group at stage A was formed:

ISO/TC 102/SC 2/WG 29A - Determination of total iron content by coulometric method.

(5). The followings are in study groups status:

(a). Uses of ICP in iron ore analyses.
(b). Determination of sources of errors in the determination of total iron content.
(c). Determination of Co content.
(d). Determination of Sn content.

MISCELLANEOUS DECISIONS

(a). The subcommittee has decided that in the future, for the purpose of international tests, the blank values will be reported for all determinations. WG 12 will be involved in the processing of these blank data.

(b). In the future the statistician report will include a recommendation on the number of significant figures the analyst must report in the routine application of the standard methods.

CONCLUSIONS

During the past period (1984/86), ISO/TC 102/SC 2 has been very active. Eight international tests have been conducted to evaluate various analytical methods. Six of these methods have been accepted in priciple as ISO standards. Again the most extensive of these tests was the determination of various elements in iron ores by wavelength dispersive X-ray fluorescence spectrometric method, conducted by WG 27A. Unfortunately, however, the main effort of this WG, i.e to develop an XRF standard method for total Fe has not been successful. The method seems to be suitable for most of the elements tried, but it is still not good enough for total iron. Due to its importance, the work in this area will be continued. Canada's contribution to the effort of this subcommittee by way of statistical analysis and data processing has been one of the major factors in the high productivity of the subcommittee. Therefore it is recommended that Canada continue her support.

RESOLUTIONS TAKEN AT THE 13th MEETING OF ISO/TC 102/SC 2 CONCERNED WITH WG 12B ACTIVITIES

Resolution 1

ISO/TC 102/SC 2 agreed to accept in principle the recommendations contained in the resolution (Annex 1) of the meeting of WG 12 held in Tokyo on 9 November 1986. (Author's note: this resolution will be annexed in the secretariat report; it contains the decision made at the WG 12 meeting).

Resolution 2

ISO/TC 102/SC 2 agreed that the mandate of WG 12 be extended for a further two years, in order to maintain the continuity of centralized data processing. The delegates also wished to record their further appreciation of the excellent work in this area being carried out by Dr. Sutarno of Canada.

Resolution 7

ISO/TC 102/SC 2 agreed to rephrase the wording of part (b) of the Note in TC 102 Chemical Analysis standards, clause 8.2.2, "Acceptance of analytical values", as follows:

(b). If the certification has been made by only one laboratory or if the interlaboratory results are missing, it is advisable not to use this material for this purpose. In case its use is unavoidable, use the formula

$$|A_c - A| \leq 2[2\,\sigma_L^2 + \sigma_r^2/n]^{1/2}$$

ISO/TC102/SC2 LIST OF ATTENDANCE 9 November 1986

Country	Name	Belonging
Australia	Mr.E.S.Pilkington	CSIRO
	Mr.M.J.Munro	SAA
	Mr.B.Ziegelaar	Mt.Newman Mining Co.
	Dr.K.Norrish	CSIRO
Brazil	Prof.Barros	UFMC
	Mr.Coutinho	USIMINAS
	Dr.Jakitsche	CVRD/SUTEC
Canada	Mr.O.Bhargava	Corporate Analytical Chemistry Stelco Inc.
	Dr.R.Sutarno	Canada Centre for Mineral & Energy Technology
	Mr.W.Van de Vrande	Dofasco Inc.
China	Mr.Zhang Jizhi	Baotou Iron and Steel Company
	Mr.Liu Changyi	China Metallurgical Standardization Reserch Institute
France	Dr.M.Jecko	IRSID
Germany	Dr.K.H.Koch	Hoesch Stahl AG
Korea	Mr.Lee Yong Mook	Sammi Company
	Mr.Cho Soung Sik	Tokyo Branch of Pohang Iron & Steel Company
Netherlands	Drs.H.Umans	Hoogovens Groep BV
Sweden	Mr.G.Airijoki	LKAB
U.K	Mr.P.Gale	British Steel Corporation
U.S.S.R	Mr.V.I.Plekhanov	Gosstandart
	Mrs.I.V.Diatchkova	〃
Japan	Dr.T.Ohtsubo(Leader)	Nippon Steel Corporation
	Mr.K.Takimoto	〃
	Mr.M.Abe	JISC
	Mr.T.Kondo	Nippon Kokan K.K.
	Mr.Y.Takano	〃
	Mr.Y.Matsumura	Kawasaki Steel Corporation
	Mr.Y.Matsumoto	Sumitomo Metal Industries,Ltd.
	Mr.M.Taniguchi	Kobelco Research Institute,Inc.
	Mr.H.Hirata	Nisshin Steel Co.,Ltd.
	Mr.Y.Imai	Japan Marine Surveyors & Sworn Measurers' Association
	Dr.S.Takaisi	Japan Iron and Steel Federation
	Mr.K.Mabuchi	〃
	Mr.T.Yoshikawa	Marubeni Coporation